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1. Title of the Invention

METHOD FOR MANUFACTURE OF
ULTRATHIN COPPER FOIL

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Specification

1. Title of the Invention

METHOD FOR MANUFACTURE OF ULTRATHIN COPPER FOIL

2. Patent Claims

A method for the manufacture of an ultrathin copper foil by conducting zinc substitution plating on the edge portion of one surface of an aluminum foil or aluminum alloy foil, then conducting copper plating to a thickness of 0.5-12 μm in a copper pyrophosphate plating bath on

the entire surface of said surface, and then conducting copper plating to a thickness of 8-12 μm in a copper sulfate-based copper plating bath on the outside thereof.

3. Detailed Description of the Invention

The present invention relates to a method for the manufacture of an ultrathin copper foil for high-density printed circuit boards in which a wiring width and wire spacing are greatly decreased.

In recent years, significant progress in electronics technology created a demand for highly reliable high-density printed circuit boards. In particular, in the copper-clad laminates for printed circuits of computers, it became necessary to form ultrafine circuits with a wiring width and wiring spacing of no more than 0.2 mm. However, the thickness of the conventionally used copper foils was as large as about 85 μm . Therefore, in the etching technology at the time, when circuits were formed from such copper foil-clad laminates, there was a danger that the copper foils would be undercut by side etching (a lower side portion of a groove is etched on the periphery thereof and a recess-like portion is obtained), causing a short circuit. For this reason, circuits with high-density wiring widths and wiring spacing could not be obtained. Therefore, it was impossible to obtain highly reliable printed circuits with a high density.

Accordingly, thin copper foils with a thickness of 18-25 μm were used to attain this object. However, since they were manufactured by electrolytic methods similar to those for the manufacture of the above-mentioned thick copper foils, pinholes easily occurred. Moreover, because the foils were so thin, "wrinkles" and "scratches" could easily occur when the foils were handled. A low production yield and a high cost were other drawbacks associated with such ultrathin foils. Furthermore, when such copper foils were employed for forming high-density circuits by a photoresist method, the lower limit for the wiring width and wiring spacing was 0.2 mm, and high-density circuits with an ultrafine pattern having wiring widths and spacing less than this limit were difficult to produce.

An improved method was suggested, this method using an ultrathin copper foil formed by employing an aluminum foil, etc. as a pseudo-substrate and electroplating a copper layer with a thickness of no more than 18 μm thereupon. A glass-epoxy base material was laminated on the copper foil surface, the aluminum foil serving as a pseudo-substrate was removed by chemical or mechanical peeling method, and the resulting copper foil laminated sheet was used to form a high-density circuit by the aforesaid photoresist process. Such a method is disclosed, for example, in Japanese Patent Application Laid-open 48-85857.

However, with such a method, it is necessary to conduct zinc substitution plating on the electrocoating surface of the aluminum foil in advance in order to provide for good bonding strength of the ultrathin copper layer formed by electroplating with the aluminum foil serving as a pseudo-substrate and it was also necessary to dissolve and remove the aluminum and zinc chemically in order to obtain a copper clad laminate after lamination of the glass-epoxy base material. Therefore, in addition to those two types being complicated and wastewater treatment being difficult, there are disadvantages in that the copper layer is partially dissolved when the zinc is removed, and circuit shorts occur easily.

With another suggested method, copper cyanide plating is conducted on one surface of the aluminum foil of the pre-degreased and activated pseudo-substrate. However, in such a case, the copper adhesion strength is insufficient. For this reason, when in subsequent processes a thick copper plating was conducted with an acidic copper plating bath and then treatment was

conducted to increase the adhesive force, the copper layer could be easily peeled off and removed from the surface of aluminum foil. Moreover, when the thickness of the copper plating was less than 12 μm , a large number of fine pinholes were present and the product was not suitable for practical use.

The inventors have conducted an intensive study aimed at the resolution of the above-described problems. Based on the results obtained, a method was developed for the manufacture of an ultrathin copper foil designed for the formation of highly reliable printed circuits with a high density. Thus, with the method in accordance with the present invention zinc substitution plating is conducted on the edge portion on one surface of an aluminum or aluminum alloy foil (referred to simply as aluminum foil hereinbelow), then copper plating is conducted to a thickness of 0.5-12 μm in a copper pyrophosphate plating bath on the entirety of that one surface, and then copper plating is conducted to a thickness of 8-12 μm in a copper sulfate-based copper plating bath on the outside thereof.

The method in accordance with the present invention will be described hereinbelow in greater detail.

At least one surface of an aluminum foil with a thickness of 30-70 μm (a long foil with a width of 300-1300 mm) is degreased with an alkaline cleaner and washed with water according to the usual procedure. If necessary, washing with water is conducted after pickling with 50% HNO_3 or activation.

Then an edge portion on one surface of the foil is immersed for 2-120 sec in the so-called zincate bath and zinc substitution plating is conducted. In this case, the edge portion as referred to hereinabove is a frame-like portion located on the periphery of the aluminum foil; it has a width of 2-100 mm, preferably, 10-50 mm. This width is referred to as an edge width.

The zinc substitution plating is conducted to improve adhesion of copper layer to the aluminum foil in the portions that are directly subjected to copper plating. Thus, when the end portion of the aluminum foil is not subjected to zinc substitution plating, a copper pyrophosphate plating itself cannot provide for a sufficient bonding strength with the surface of aluminum foil. When, in such a state, water washing and copper sulfate thick plating are performed in the subsequent process, the copper layer can easily peel from the edge portion of the aluminum foil. Moreover, if the coating peels off even slightly, then water or plating solution rapidly penetrates into the interface between the aluminum foil and copper layer, the surface of aluminum foil is corroded mainly by the plating solution, and the peeling is further promoted. Even if the substrates in such a state are finally dried, since the major portion of the copper layer has peeled from the pseudo-substrate, "wrinkles" or a large number of pinholes appear therein, making the foil unsuitable for practical use. However, if zinc substitution plating of the edge portion of aluminum foil is conducted as described above, the bonding strength with respect to the copper pyrophosphate plating bath is also improved and no peeling occurs in subsequent plating in a copper sulfate bath. Moreover, when the copper plating is formed to a thickness of no less than 5 μm , practically no pinholes are formed.

Immediately after completion of the zinc substitution plating, a copper layer with a thickness of 0.5-12 μm is plated by using a copper pyrophosphate plating bath, followed by washing with water. In such a case, no specific limitation is placed on the composition of the copper pyrophosphate plating bath, but it is preferred that the copper concentration be 5-100 g/L, the concentration of potassium pyrophosphate be 100-500 g/L, and the pH be 8-12. As for the plating conditions, the bath temperature is within a range from normal temperature to 70°C, the electric current density is 1-5 A/dm^2 , and the electrolysis time is 5-25 sec.

The copper pyrophosphate plating bath is selected for the purposes of the present invention because the coating obtained with such a bath has better adhesion to aluminum foils than that obtained with other copper plating baths, and, naturally, no peeling occurs in the lamination of laminated base materials for printed circuits. Furthermore, in addition to that fact that stronger adhesion cannot be maintained, it is not necessary to chemically dissolve and remove the aluminum foil pseudo-substrate after lamination of the base material, and peeling can be conducted by a simple mechanical means. As a result, wastewater treatment is made unnecessary, and the aluminum used can be easily recovered in the form of aluminum chips. Furthermore, a copper pyrophosphate plating bath makes it possible to conduct uniform electrocoating, and a coating can be obtained which is smooth and has good gloss. Moreover, a very small number of pinholes appear even when a thin copper foil is obtained. The thickness of the copper pyrophosphate plating is limited to 0.5-1.2 μm for the following reasons. When the thickness is less than 0.5 μm , the number of pinholes in a thin copper foil obtained increases, making it unsuitable for practical use. On the other hand, when the thickness exceeds 12 μm , since the upper limit of thickness of thin copper foils is 15 μm , the copper sulfate plating in the subsequent process cannot be conducted. Moreover, adhesion to plastic laminated base materials is degraded. The thickness of the copper foil layer is preferably 2-7 μm .

Plating copper to a thickness of 8-12 μm in a copper plating bath containing copper sulfate is conducted as a method for roughening of the copper foil layer. Appropriate conditions of such treatment should be selected and no specific limitation is placed thereon. Typically the copper plating bath has a copper concentration of 20-60 g/L and a sulfuric acid concentration of 40-150 g/L. The bath temperature is preferably within a range from normal temperature to 60°C. As for the electric current density and electrolysis time, they are somewhat affected on the liquid flow parameters, stirring state, and plating thickness, but preferably they are usually within the ranges of 5-40 A/dm² and 1-10 min, respectively. The external appearance of the obtained copper plated film must be such that velvety peaks and valleys of red color are formed.

The thickness of the copper plated layer is limited to 8-12 μm for the reasons as follows. The entire thickness of plated copper layer may be 15 μm and the thickness of the plated layer obtained by using a copper sulfate bath may be adjusted by varying the thickness of the plated layer obtained with the above-mentioned copper pyrophosphate plating bath.

The plated layer with the above-mentioned roughening treatment is further laminated with a plurality of layers of a composition prepared by infiltrating an insulating substance such as glass fibers with an epoxy resin, and then the aluminum foil pseudo-substrate is peeled off, thereby obtaining a final product.

In this case, the bonding strength between the plated copper layer subjected to the roughening treatment and said laminated insulating material may be about 1.6-1.8 kg/cm², but if it is less than 1.6 kg/cm², secondary roughening treatment copper plating is conducted on the outer side of the copper plated layer. For this purpose a method can be used in which, for example, cathodization is conducted at a current density of 2-20 A/dm² for 2-120 seconds in an acidic copper electrolytic bath having added therein ions of at least one metal selected from a group including arsenic, antimony, and bismuth.

Embodiments of the present invention will be described hereinbelow

Embodiment 1

A soft pure aluminum foil (99.9%) with a thickness of 70 μm was degreased by immersion for 3 min in a solution of sodium orthosilicate (100 g/L) at a temperature of 70°C, and then pickling and activation treatment was conducted by immersion for 1 min at a temperature of 25°C in an aqueous solution of HNO_3 with a concentration of 350 g/L. Then, an edge portion (edge width 20 mm) on only one side of the aluminum foil was selectively subjected to local zinc substitution plating by immersion for 60 sec in a solution (liquid temperature, room temperature) consisting of ZnO 30 g/L and NaOH 100 g/L. Then, one side of the aluminum foil was plated with copper for 5 min at an electric current density of 2 A/dm^2 in a plating bath (liquid temperature 50°C) having a Cu concentration of 30 g/L and a potassium pyrophosphate concentration of 150 g/L and a pH of 8.8. Finally, the plated layer was subjected to roughening copper plating for 2 min at a current density of 10 A/dm^2 in a plating bath (liquid temperature 20°C) containing Cu at 20 g/L and H_2SO_4 at 75 g/L. As a result, an ultrathin copper foil with a copper layer thickness of 6 μm was obtained.

Embodiment 2

A hard aluminum alloy foil (Al 99.7%, Cu 0.05%, Fe 0.05%, Si 0.20%) with a thickness of 30 μm was subjected to degreasing by immersion for 30 sec at a temperature of 50°C in a solution containing sodium orthosilicate 100 g/L, NaOH 30 g/L, and nonionic surfactant 0.1 g/L. Then, pickling and activation treatment was conducted by immersion for 30 sec at a temperature of 25°C in a solution consisting of HCl 100 g/L and NaCl 50 g/L. Then, local zinc substitution plating was conducted selectively only on the edge portion (edge thickness 35 mm) on one side of the aluminum alloy foil by immersion for 38 sec in a solution (liquid temperature, room temperature) consisting of ZnO 5.0 g/L, NaOH 200 g/L, and FeCl_3 5 g/L. One surface of the aluminum foil was then plated with copper for 10 min at a current density of 3 A/dm^2 in a plating bath (liquid temperature 60°C) consisting of Cu 80 g/L and potassium pyrophosphate 30 g/L, and having a pH of 9.3. Then roughening copper plating of said plating layer was conducted for 60 sec at a current density of 6 A/dm^2 in a plating bath (liquid temperature 25°C) consisting of Cu at 30 g/L and H_2SO_4 at 100 g/L. Finally, the resulting roughened plated layer was subjected to secondary roughening copper plating for 20 sec at a current density of 6 A/dm^2 in a solution consisting of $\text{Cu}(\text{BF}_4)_2$ 25 g/L, HBF_4 42 g/L, $\text{Na}_3\text{AsO}_4 \cdot 12\text{H}_2\text{O}$ 0.9 g/L (liquid temperature 12°C), and an ultra thin copper foil with a copper layer thickness of 7 μm was obtained.

Embodiment 3

A hard aluminum alloy foil (Al 99.3%, Cu 0.04%, Fe 0.07%, Si 0.15%, Zn 0.05%, Mg 0.10%, Ti 0.05%) with a thickness of 50 μm was subjected to anodic electrolytic degreasing for 30 sec at a current density of 2 A/dm^2 in a solution containing sodium orthosilicate 100 g/L, NaOH 30 g/L, and a nonionic surfactant 0.1 g/L. Then only the edge portion (edge width 15 mm) of the Al alloy foil was selectively subjected to local zinc substitution plating by treating for 20 sec in a solution (liquid temperature 30°C) consisting of ZnO 70 g/L and NaOH 400 g/L. Then, one surface of the aluminum foil was plated with copper for 15 minutes at a current density of 3 A/dm^2 in a plating bath (liquid temperature 60°C) having a Cu concentration of 100 g/L, a potassium pyrophosphate concentration of 250 g/L, and a pH of 9.0. Then, the copper plated

layer was subjected to roughening copper plating for 75 sec at a current density of 32 A/dm² in a plating bath (liquid temperature 65°C) having a Cu concentration of 50 g/L and an H₂SO₄ concentration of 50 g/L. Finally, the roughened copper plated layer was subjected to secondary roughening copper plating for 32 sec at an electric current density of 4 A/dm² in a solution (liquid temperature 15°C) containing Cu at 7 g/L, H₂SO₄ at 32 g/L, H₃AsO₄ at 0.4 g/L, and H₂SiO₃ at 0.2 g/L, and an ultrathin copper foil with a copper layer thickness of 15 µm was obtained.

Comparative Example 1

A hard aluminum alloy foil (Al 99.7%, Cu 0.5%, Fe 0.05%, Si 0.20%) with a thickness of 30 µm was subjected to degreasing by immersion for 3 min in a solution (liquid temperature 70°C) consisting of sodium carbonate 30 g/L and sodium phosphate 50 g/L, and then an edge portion of 15 mm on one surface of the aluminum alloy foil was selectively subjected to zinc substitution plating by immersion for 20 sec in a solution containing ZnO at 70 g/L and NaOH at 400 g/L (liquid temperature 30°C). Then, copper plating was performed on the entirety of one surface of the aluminum alloy foil for 15 minutes at an electric current density of 3 A/dm² in a plating bath (liquid temperature 40°C) having a Cu concentration of 100 g/L, a potassium pyrophosphate concentration of 250 g/L, and a pH of 9.0, then the copper plated layer was subjected to roughening copper plating for 75 sec at an electric current density of 32 A/dm² in a plating bath (liquid temperature 65°C) having a Cu concentration of 50 g/L and an H₂SO₄ concentration of 50 g/L. Finally, the roughened copper plated layer was subjected to secondary roughening copper plating for 32 sec at an electric current density of 4 A/dm² in a solution (liquid temperature 15°C) containing Cu at 7 g/L, H₂SO₄ at 32 g/L, H₃AsO₄ at 0.4 g/L, and H₂SiO₃ at 0.2 g/L, and an ultrathin copper foil with a copper layer thickness of 5 µm was obtained.

Comparative Example 2

An aluminum alloy foil identical to that of Comparative Example 1 was used and degreasing was conducted in the same manner as in Comparative Example 1. Then, copper plating similar to that of Comparative Example 1 was conducted immediately without zinc substitution plating and thereafter roughening copper plating was conducted to obtain an ultrathin copper foil with a copper layer thickness of 5 µm.

The ultrathin copper foils with pseudo-substrates attached thereto, which were obtained in the Embodiments 1-3 and Comparative Examples 1-2 were laminated on base materials infiltrated with glass epoxy and then pressing was conducted for 60 min at a pressure of 25 kg/cm² and a heating temperature of 160°C to obtain a laminated sheet with a thickness of 2 mm. Copper-clad laminated sheets were then obtained by mechanically peeling off the pseudo-substrates of aluminum foils, copper pyrophosphate plating was thereafter conducted to increase the thickness of the copper layer to 15 µm, and the bonding strength and pinholes were measured by the usual method. The results are presented in Table 1.

The bonding strength was measured by applying a peeling force in the direction of 90° to a width of 10 mm. The number of pinholes was counted by illuminating the layer from below a dark room and observing the transmitted light.

Table 1.

	Number of pinholes (holes/m ²)	Bonding strength (kg/cm ²)	External appearance of copper foil
Embodiment 1	2-4	1.6-1.7	Fine velvety peaks and valleys
Embodiment 2	1-2	2.0-2.4	"
Embodiment 3	0	2.0-2.4	"
Comparative Example 1	20-100	1.5-1.7	Bulbous peaks and valleys
Comparative Example 2	Very large number	1.4-1.8	"

As follows from the table presented above, the ultrathin copper foils obtained by the method in accordance with the present invention have a number of pinholes substantially less than that in the ultrathin foils obtained by the conventional methods and also have excellent bonding strength.

Patent Representative. Patent Attorney: Takehiko Suzue.

5. List of Appended Items

(1) Power of attorney	2 copies
(2) Specification	1 copy
(3) Drawings	1 copy 1 [illegible]
(3) (4) Copy of application	1 copy 3 [illegible]

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特開52-16433 (中)

Cu 0.03 g, Fe 0.03 g, Bi 0.20 g) をアルトケイ酸ナトリウム 100 g/g, NaOH 30 g/g, ノニオン系界面活性剤 0.1 g/g からなる溶液中に 50℃ において 30 秒浸漬して脱脂を行い、次いで HCl 100 g/g, NaCl 50 g/g からなる溶液中に 25℃ において 30 秒浸漬して脱洗剤に感化処理を行った後、該アルミニウム合金箔の片面酸化膜厚 (膜厚 3.5 nm) のみを選択的に ZnO 50 g/g, NaOH 200 g/g, FeCl_3 5 g/g からなる溶液 (液温、室温) 中に 30 秒間浸漬せしめ部分蒸着膜メッキを行った後、次いで該アルミニウム箔の片面を Cu 80 g/g, ビニロン酸カリウム 30 g/g, pH 9.3 からなるメッキ液 (液温 60℃) 中において 10 分間、電流密度 3 A/dm² によりメッキを行い、次いで該メッキ層を Cu 濃度 30 g/g, H_2SO_4 100 g/g からなるメッキ液 (液温 25℃) 中において 60 秒間、電流密度 6 A/dm² により凹凸化メッキを行い、最後に該凹凸化メッキ層の上に $\text{Co}(\text{BF}_4)_2$ 25 g/g, HEP, 43 g/g,

$\text{Na}_2\text{AsO}_4 \cdot 12\text{H}_2\text{O}$ 0.9 g/g からなる溶液 (液温 12℃) 中において 20 秒間、電流密度 6 A/dm² にて 2 次凹凸化メッキを行って銅厚 7 μm の複層銅箔を得た。

実施例 3

50 μm の厚さアルミニウム合金箔 (AL99.99, Cu 0.04 g, Fe 0.07 g, Bi 0.15 g, Zn 0.03 g, Mg 0.10 g, Ti 0.05 g) をアルトケイ酸ナトリウム 100 g/g, NaOH 30 g/g, ノニオン系界面活性剤 0.1 g/g からなる溶液中に 30 秒間電流密度 2 A/dm² により脱脂処理を行い、次いで該 AL 合金箔の片面 (膜厚 1.5 nm) のみを選択的に ZnO 70 g/g, NaOH 400 g/g から脱脂 (液温 30℃) 中に 20 秒間浸漬せしめて部分蒸着膜メッキを行った後、次いで該アルミニウム箔の片面を Cu 濃度 100 g/g, ビニロン酸カリウム 250 g/g, pH 9.0 からなるメッキ液 (液温 60℃) 中において 15 分間、電流密度 3 A/dm² にてメッキを行い、次いで該メッキ層

を Cu 濃度 50 g/g, H_2SO_4 30 g/g からなるメッキ液 (液温 65℃) 中において 75 秒間、電流密度 3.2 A/dm² にて凹凸化メッキを行い、最後に該凹凸化メッキ層の上に Cu 濃度 7 g/g, H_2SO_4 32 g/g, H_2AsO_4 0.4 g/g, H_2SiO_3 0.2 g/g からなる溶液 (液温 15℃) 中において 32 秒間、電流密度 4 A/dm² にて 2 次凹凸化メッキを行って銅厚 1.5 μm の複層銅箔を得た。

比較例 1

30 μm の厚さアルミニウム合金箔 (AL99.75, Cu 0.5 g, Fe 0.05 g, Bi 0.30 g) を炭酸ソーダ 30 g/g, リン酸ソーダ 50 g/g からなる溶液 (液温 70℃) 中に 3 分間浸漬して脱脂を行い、次いで該アルミニウム合金箔の片面膜厚 1.5 nm を選択的に ZnO 70 g/g, NaOH 400 g/g からなる溶液 (液温 30℃) 中に 20 秒間浸漬して部分蒸着メッキを行い、次いで該アルミニウム合金箔の片面の全面を Cu 濃度 100 g/g, ビニロン酸カリウム 250 g/g,

pH 9.0 からなるメッキ液 (液温 40℃) 中に 15 分間、電流密度 3 A/dm² にてメッキを行い、次いで該メッキ層の上に Cu 濃度 30 g/g, H_2SO_4 50 g/g からなるメッキ液 (液温 65℃) 中において 75 秒間、電流密度 3.2 A/dm² にて凹凸化メッキを行い、最後に該凹凸化メッキ層の上に Cu 濃度 7 g/g, H_2SO_4 32 g/g, H_2AsO_4 0.4 g/g, H_2SiO_3 0.2 g/g からなる溶液 (液温 15℃) 中において 32 秒間、電流密度 4 A/dm² にて 2 次凹凸化メッキを行って銅厚 5 μm の複層銅箔を得た。

比較例 2

比較例 1 と同様のアルミニウム合金箔を使用し、比較例 1 と同様に脱脂処理を行った後、部分蒸着メッキを行うことなく、直ちに比較例 1 と同様のメッキ液を行い、更に凹凸化メッキを行って銅厚 5 μm の複層銅箔を得た。

而して上記実施例 1～3 及び比較例 1～2 により得た複層体付銅箔をガラスエポキシ樹脂層の上に重ねた後、加熱温度 150℃、圧

特許第16433号

力2.5kg/cm²において60分間加圧して、厚さ2mmの積層板とした後、アルミニウム箔の板層と機械的に引を制して剥貼積層板とした後、積層の厚さを1mmにするため、更にビニル被膜ノリをを行い、両面により粘着力をピンホールを決定した。その結果は図1表に示す通りである。

なお、粘着力は10mm幅につき90°方向の引を利し力によるものであり、ピンホールは場所において下方から光をあて透過する穴の数をカウントした。

本特許は従来方法により得た積層板に比してピンホールが著しく少く且つ粘着力において優れたものを得る効果的な効果を得る。

出願人代理人 弁護士 鈴木 武 彦

図 1 表

	ピンホール数 (個/cm ²)	粘 着 力 (kg/cm ²)	積層の外観
電線銅1	2~4	1.5~1.7	銀箔ビロード状凹凸
2	1~2	2.0~2.4	“
3	0	2.0~2.4	“
比較例1	20~100	1.5~1.7	ダング状凹凸
2	無 数	1.4~1.8	“

上表より明かの如く本発明方法により得た積

5. 添付書類の目録

(1) 書 件 名 2通
 (2) 特 許 書 1通
 (3) 特 許 料 1通 (1万円)
 (4) 特 許 料 1通 (1万円)

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